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<b>(21) International Application Number:</b> PCT/US96/20453 <b>(22) International Filing Date:</b> 13 December 1996 (13.12.96) <b>(30) Priority Data:</b> 08/573,043 15 December 1995 (15.12.95) US <b>(71) Applicant:</b> POWER PLANT AGGREGATES OF IOWA, INC. [US/US]; 2220 Hawkeye Drive, P.O. Box 355, Sioux City, IA 51102 (US). <b>(72) Inventors:</b> GREENE, Gary, L.; Power Plant Aggregates of Iowa, Inc., 2220 Hawkeye Drive, P.O. Box 355, Sioux City, IA 51102 (US). JENSEN, Mark, R.; Power Plant Aggregates of Iowa, Inc., 2220 Hawkeye Drive, P.O. Box 355, Sioux City, IA 51102 (US). JORGENSEN, Michael, D.; Power Plant Aggregates of Iowa, Inc., 2220 Hawkeye Drive, P.O. Box 355, Sioux City, IA 51102 (US). ZIMMERMAN, Lonnie, G.; Power Plant Aggregates of Iowa, Inc., 2220 Hawkeye Drive, P.O. Box 355, Sioux City, IA 51102 (US). <b>(74) Agent:</b> ZARLEY, Donald, H.; Zarley, McKee, Thomte, Voorhees & Sease, Suite 3200, 801 Grand Avenue, Des Moines, IA 50309-2721 (US).		<b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> METHOD OF STABILIZING ODORS IN MANURE		
<b>(57) Abstract</b>		
A process of stabilizing odors in animal manure comprised of water and solids by adding an admixture thereto to adjust the pH of the manure to a level that will minimize the release of odorous compounds including ammonia and hydrogen sulfide to the surrounding air.		

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TITLE: METHOD OF STABILIZING ODORS IN MANURE

BACKGROUND OF THE INVENTION

The treating of animal and human wastes for various purposes has been taking place for centuries. In modern times, this activity has centered primarily on municipal sewage and waste water treatment plants, and on manure collected at livestock and poultry feeding facilities. In regard to municipal treatment plants, much attention has been directed to dewatering the sewage and then adding various alkaline materials to the resulting sludge to raise the pH of the mixture to 12 and above. This is done to stabilize the pathogenic bacteria whereupon the sludge is more suitable for use as fertilizer and is environmentally acceptable. Regulations of the United States Environmental Protection Agency (EPA) closely limit this activity. Examples of this technology are shown in U.S. Patents Nos. 5,277,826; 4,902,431; 4,781,842 and 4,554,002.

In the area of animal and poultry wastes, the odor emitted by the manure, has been the primary concern, and while this matter has been addressed (e.g. U.S. Patent No. 4,902,431), no universally acceptable manure odor stabilizing processes have been developed to effectively combat this problem. Animal and poultry wastes appear in different locations and different holding devices in a multitude of animal and poultry confinement buildings which have emerged in great numbers over the last 25 years. These facilities include slotted floors and solid floors from which manures are collected by manual or mechanical scraping or flushing; gutters; recirculation flush pits; and gravity flow channels. They also include open feed lots utilizing paved or earthen surfaces with runoff channels of varying designs. Liquid-solid separation systems include settling tanks, basins, channels, mechanical separation systems, evaporation ponds and dehydrators. Liquid manure storage systems utilize manure pits, earthen storage basins (i.e., lagoons), and aboveground

tanks.

It is therefore a principal object of this invention to provide a method of stabilizing odors in manure, and particularly animal manure, wherein the manure is comprised of liquid and solids.

A further object of this invention is to provide a process of stabilizing odors in manure which will be effective in treating the manure in a large variety of collection devices.

A still further object of this invention is to provide a method of stabilizing odors in manure which is effective in manures of differing composition.

A still further object of this invention is to provide a method of stabilizing odors in manure which will preserve the fertilizer potential of the manure.

A still further object of this invention is to provide a method of stabilizing odors in manure which is economical and cost-effective.

A still further object of this invention is to provide a method of stabilizing odors in manure which will be environmentally acceptable.

These and other objects will be apparent to those skilled in the art.

## SUMMARY OF THE INVENTION

This invention involves the method of adding lime, fly ash, cement kiln dust or the like, or mixtures thereof, with manure comprised of liquid and solids in sufficient quantity to raise the pH thereof to a minimum of 7, a maximum level of 10.5, and an optimum of 9.5 to minimize the release of ammonia, hydrogen sulfide, and other odor producing elements from the manure.

## BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a bar graph showing the change in odor threshold versus time after start up for three levels of alkaline addition versus an untreated control. The levels of

treatment were 5 lb, 2.5 lb, and 1.25 lb additive added per 1 lb of solids in the manure;

Fig. 2 is a bar graph showing the change in pH of treated manure versus time after start up for three levels of alkaline addition versus an untreated control. The levels of treatment were 5 lb, 2.5 lb, and 1.25 lb additive added per 1 lb of solids in the manure;

Fig. 3 is a bar graph showing the change in ammonia levels of the headspace of a manure storage tank versus time after start up for three levels of alkaline addition versus an untreated control. The levels of treatment were 5 lb, 2.5 lb, and 1.25 lb additive added per 1 lb of solids in the manure;

Fig. 4 is a bar graph showing the pH values of five treated samples of manure compared to the untreated control sample with 0.25 lbs of additive added per 1 lb of solids in the manure;

Fig. 5 is a bar graph showing the pH values of five treated samples of manure compared to the untreated control sample with 0.5 lbs of additive added per 1 lb of solids in the manure;

Fig. 6 is a bar graph showing the odor intensity values of five treated samples of manure compared to the untreated control sample with 0.25 lbs of additive added per 1 lb of solids in the manure;

Fig. 7 is a bar graph showing the odor intensity values of five treated samples of manure compared to the untreated control sample with 0.5 lbs of additive added per 1 lb of solids in the manure;

Fig. 8 is an elevational view of one of the laboratory test apparatus used to determine the utility of this invention.

Fig. 9 is a bar graph showing the odor threshold from the liquid surface of treated samples compared to an untreated sample as contained in earthen storage at various times of the year; and

Fig. 10 is a bar graph showing the change in odor

threshold from the liquid surface of treated samples contained in concrete storage versus time.

#### DESCRIPTION OF THE PREFERRED EMBODIMENT

5       The purpose of this invention is to add sufficient materials, primarily alkaline materials, to manure, comprised of both liquid and solids, to raise the pH thereof to an optimum level of 9.5 which will minimize the release of ammonia and hydrogen sulfide and other odor producing gases.

10      The materials to be used as the additive to the manure are lime, kiln dust, fly ash, or derivatives thereof, including mixtures of these ingredients and other materials derived from calcining processes, combustion by-products and powdered adsorbents containing activated carbon and/or dry clay. The

15      major constituents of cement kiln dust and fly ashes are oxides of calcium, silica, aluminum, and sulfur.

      The process of this invention can be conducted wherever the animal manure is collected. However, manures collected in pits, holding tanks or earthen basins are most convenient for

20      implementation of the process. While the process is useful for the treatment of any type of waste, it is particularly suitable for the treatment of animal wastes, especially in animal and poultry confinement operations. The term "animal wastes" as used herein will be understood to include poultry

25      manure as well as animal manure.

      The principal thrust of this invention is to treat the animal wastes so as to settle the solids in the manure with respect to the liquid portion thereof, and to adjust the pH of the manure, and particularly the liquid portion thereof to

30      a level that will minimize the release of the odorous compounds from the manure. Specifically, the pH is adjusted to an optimum level of 9.5 which will minimize the release of both ammonia and hydrogen sulfide which are two of the principal odor causing factors. This technique also is

35      believed to suppress the release of other odor causing compounds. By suppressing the release of ammonia, the use of the treated manure as fertilizer is substantially enhanced.

The admixture used for increasing the pH of manure in its natural state (normally in the range of 6.0 to 7.0) is usually, but not always, an alkaline material comprised of one or more of lime, kiln dust or fly ash, or derivatives thereof. Kiln dust and fly ash are plentiful and generally less expensive than lime.

The admixtures are preferably added to the manure at the rate of 0.2 to 1 lb of admixtures to 1 lb of manure, respectively, by dry weight of solids. They can be added by special equipment as described in the Field Test set forth hereafter, or through any convenient material handling system. The admixture is added periodically to the manure to maintain the pH thereof within the above defined range. Thorough agitation of the mixture of manure and additives to provide a homogenous mixture is required in all applications. Under some conditions, daily applications may be necessary. Daily checking of pH values is not necessary, but may be required if problems are encountered.

Alkaline by-products from power plants, portland cement plants, and lime manufacturing plants were evaluated to determine the effectiveness of controlling swine odors from manure storage. Five products were tested in the laboratory and one of the products was chosen to be further evaluated in the field. The odor evaluation was done with an olfactometer using the triangular-forced-choice method. The study showed that odors are substantially reduced using all the byproducts tested in the laboratory. The cement kiln dust was chosen for the field study.

Swine manure storage facilities are the major potential odor sources from swine operations. Alkaline treatment of manure can help reduce odors from the manure storage and during land application. When the waste is collected and stored, it undergoes decomposition due to the metabolic actions of microorganisms. The manure gases of odor concern are ammonia, hydrogen sulfide, and volatile odorous compounds.

EXAMPLE 1

## Pilot Study

This pilot study utilized the alkaline byproduct, cement kiln dust (CKD) and manure from the Iowa State Nutrition Research Facility. The CKD was provided by conventional feed truck fed into a CementTech LSP-3000 liquid sludge processor (see U.S. Patent No. 5,284,578).

The initial rates of CKD to manure on a dry weight basis were 2.3 lb to 1 lb, and 0.5 lb to 1 lb. The manure used in the field test had an actual solids content of 0.8 percent. Adjusted addition rates based on the actual solids content were 5 lb, 2.5 lb and 1.25 lb additive to 1 lb manure solids.

Odor threshold is the ratio of the number of volumes of fresh air required to mix with one volume of odorous air so that the odor can hardly be detected. CKD addition to manure provided significant odor reduction during the mixing, storage and final land application of the manure used in this field test. The manure used in all four treatments was agitated thoroughly just prior to air quality sample collection on day 36 of the trial. Analysis of the final sample showed an odor threshold of 1250 for the untreated control compared to 59,107, and 362 for treatments 1, 2 and 3, respectively. Detectable odor levels were effectively reduced by 95 percent, 91 percent, and 71 percent at the three treatment levels compared to the untreated control. The treated manure in the pilot test was agitated and field applied on day 42 of the trial. Air samples were taken from the application sites and analyzed for odor thresholds. Low odor thresholds (less than 8) were found for all treatments.

It was determined that the addition of the CKD material caused the solids in the liquid manure to settle. This settling phenomenon is very important because it allows a watercap to form over the settled solids. This watercap provides a physical barrier between the odorous solid material and the atmosphere. The watercap also minimizes the oxygen content in the solid material which effectively inhibits the bacterial activity in the solids which in part



is responsible for generation of odorous gases. The CKD also increases the pH level of the liquid existing above the solids to optimum pH levels of 9.5 which suppresses the production of hydrogen sulfide gas and aids in minimizing the release of ammonia as well as other odor producing gases.

#### EXAMPLE 2

##### Laboratory Study

The laboratory study used five different alkaline byproducts including combustion residues (fly ashes), lime kiln dusts and cement kiln dusts. Calcium oxide was a major oxide component in each of the alkaline byproducts used. The manure for the experiment was from an 1100 head swine finishing building. The manure was collected from a small pit at the end of the building into which the manure was scraped as needed, generally, twice daily. The manure was agitated prior to collection for treatment in the laboratory.

Twelve plastic columns of the type Shown in Fig- 8 were fifteen inches in diameter by 48 inches high and were filled to a level of thirty six inches with manure and the alkaline byproduct. The material was mixed with the manure prior to filling the columns. Two levels of addition of five different alkaline byproducts, 0.25 lb and 0.5 lb added to one lb of solids in the manure, and one untreated control were used in the experiment. The manure had a solids content of 4.7 percent.

Each column was sealed and one-half cubic feet per minute of air was continuously pumped into the column headspace and exhausted to the outside. The air in the room surrounding the tanks was maintained between 65 to 70 degrees Fahrenheit.

Air samples were taken from the headspace on day 3, 10, and 21 to evaluate the odor, ammonia and hydrogen sulfide level. Liquid sampling included tests for pH, TNP (CCE), Potassium, Nitrogen (Total), Nitrogen (Ammonia), Nitrate +Nitrite and Phosphorus.

Figures 4 and 5 show the change of pH with time for each product. As the treatment level went from 0.25 lb to 0.5 lb

of product per lb of solids in the manure, the pH increased. The manure without treatment (6) had the lowest pH of approximately 7.0. The pH basically increased to the highest level at day 10 and decreased back to a slightly lower level at day 21 for the 0.25 lb of additive per lb of solids in the manure. Figure 5 shows the pH was between 8.5 to 9.2 for the products at a level of 0.5 lb of additive per lb of solids in the manure. The pH remained almost constant throughout the total test period.

The ammonia levels in the headspace of the columns were higher than the control in all cases. It can be observed that the pH at the lower level of additive resulted in 2 to 5 times higher ammonia level than the control. The higher level of product resulted in 4 to 17 times higher ammonia level.

Table 1. Average ammonia level in headspace of columns.

Treatment	Level of Treatment	Level of Treatment
	0.25 LB/solids (NH3 ppm)	.50 LB/solids (NH3 ppm)
1	11.5	61
2	14.5	38
3	11.5	17
4	15.5	24
5	7.0	12
6 (control)	3.5	3.5

The odor levels in Figures 6 and 7 show the decrease of odor threshold as the experiment is conducted. The figures show the reduction of odors to that on day three. Figure 7 shows that the additional additive continues to reduce the odors generated in most cases. The control shows very little change in odor level during the entire study.

Table 2 shows more variability within each product level than between the levels of product added. The control is also in the middle of the range in all chemicals evaluated. The chemical analysis for two of the 0.25 lb products added per

1 lb of manure solids was not analyzed.

Table 2. Chemical analysis of manure samples from the columns at the end of the experiment.

5

No.	Chemical Components				
	K %	N(T) %	N(A) %	NOx %	P %
2A	0.36	0.39	0.28	7.9	0.26
3A	0.25	0.41	0.30	5.8	0.24
4A	0.23	0.38	0.30	9.4	0.14
6A	0.28	0.42	0.30	5.5	0.27
1B	0.24	0.42	0.30	8.5	0.24
2B	0.29	0.43	0.27	6.0	0.29
3B	0.24	0.44	0.27	5.6	0.28
4B	0.29	0.43	0.30	8.0	0.26
5B	0.23	0.43	0.31	6.6	.028

Note: 2A to 5A is at the 0.25 LB additive  
 6A is the control  
 1B to 5B is at the 0.5 LB additive in the same order  
 as the product numbers  
 K (potassium)  
 N(T) (total nitrogen)  
 N(A) (ammonia nitrogen)  
 NOx (nitrate-nitrite)  
 P (phosphorus)

10

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### EXAMPLE 3

#### 20 Field Study

The same 1100 head finishing building from which manure was collected for the previously described laboratory study was utilized for this Field Study. This production facility was equipped with an under-slat scraping system and an earthen manure storage basin. The equipment utilized in the process was installed at the site in the spring of 1995. The equipment included a 4 ton silo for alkaline byproduct

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storage, a volumetric metering device with electronic programmable controller, a 3,000 gallon manure mixing tank, and an agitator pump to mix the product in the manure and to pump the treated manure into the earthen basin.

5       The building was scraped twice daily and the manure gravity flowed from the swine building to the 3,000 gallon buried concrete mixing tank. The operator measured the depth of the manure in the tank and entered the mass of the alkaline byproduct into the controller based upon the manure  
10 volume. The remainder of the processing of the material was automatic based upon preset parameters. The mixing pump was set to operate for 3 minutes after the material was augered into the underground manure tank and then the treated manure was pumped into the earthen basin.

15       The alkaline byproduct utilized in the field study was the product 4 analyzed in the laboratory study. Initial addition rate of alkaline byproduct was .25 lb to 1 lb of dry manure solids. When the project was initiated, it was estimated that the earthen basin contained approximately  
20 240,000 gallons of manure. Alkaline byproduct (see above) was added to the basin on June 7, 1995 by complete agitation of the basin through the 3,000 gallon mixing tank to bring the ratio of alkaline byproduct to manure solids in the earthen basin to the desired treatment level.

25       The production facility utilized as the untreated control was selected as it was similar to the treated facility. The age, gender, and genetics of the hogs and the feeding rations used at both facilities were close to identical. The control facility was located within 20 miles  
30 of the treated facility.

      The data from the treated earthen basin (Figure 9) showed that the odor generation was significantly higher during the first part of the summer. These results led to a decision to increase the addition ratio to .3 lb alkaline  
35 material to 1 lb manure solids in early July. It was also discovered by depth measurement that we had drastically underestimated the amount of manure in the basin. The

corrected dose rate of alkaline material was added to the earthen basin on July 11, 1995.

As the field study progressed, the reduction in odor was shown to continue to increase with time. The control unit had significantly higher odor levels than the treated unit. The normal flow of material added on a day to day basis was at the rate of 0.3 lb per lb of solids. The solids was based upon 4 percent solids to account for using spray water for cooling the pigs during warm weather.

The gases produced from the surface of the earthen basin were evaluated for odor, ammonia, and hydrogen sulfide. Liquid samples were taken and similar analysis to the laboratory study was made. Downwind odor evaluation at the road was measured with a scentometer.

The odor level was significantly lower when standing on the downwind side of the earthen basin with treatment. The graph in Figure 9 shows that the odor was approximately 55 to 60 percent less in the treated slurry than in the control.

The odor level on September 10 is shown to be as strong as on the control, but lower than typical for the control unit. It was observed that a change in weather accompanied that particular day. The same observation was made on another day of similar climatic conditions; however the odor level was not as strong as on the control slurry basin.

The odor downwind was significantly affected by the treatment of the slurry. Often times, the odor from the treated manure could not be detected at a distance of 500 feet from the basin.

The following observations and conclusions can be drawn from the study:

- The odor level can be significantly reduced by using alkaline byproducts at the levels tested.
- The effect of the nutrient value of the manure was less than the variation between the samples.
- The downwind odor level was significantly reduced by the use of the product.

EXAMPLE 4

## Field Study

In this field study manure was treated from a 1,500 head finishing facility. This system utilized slatted floor, scraper and pull plug to transfer manure to a 600,000 gallon cylindrical concrete holding basin. Equipment utilized on this site included a tractor (PTO) driven agitation pump to homogeneously blend the alkaline byproduct with the stored manure. The alkaline byproduct used in this study was a fluidized bed fly ash. The fly ash was delivered to the site by a pneumatic tanker truck and transferred to a mobile feeding system. This mobile feeding system delivered the alkaline byproduct to the manure storage tank at a ratio of 0.3 lb alkaline byproduct to 1 lb of manure solids.

There was approximately 360,000 gallons of manure in the storage basin prior to alkaline treatment. Approximately 80,000 gallons of fresh manure was added to the basin simultaneously with the addition of the alkaline byproduct. Solids concentration of the manure was estimated to be 4.00%. Mixing was initiated prior to addition of alkaline byproduct to the basin, continued during addition, and extended for a time period following completion of addition to ensure homogeneity of the mixture.

The gases produced from the surface of the concrete basin were evaluated for odor, ammonia, and hydrogen sulfide. Liquid samples were taken and similar analysis to the laboratory study was made. Downwind odor evaluation at the road was measured with a scentometer.

The data from the concrete storage basin (Figure 10) showed that the odor generation was significantly higher before alkaline treatment. The reduction of odor detected immediately after treatment was maintained during the extended storage time with no agitation. Reduced odor levels were maintained during agitation, prior to, and during field application.

The odor level was significantly lower when standing on

the downwind side of the concrete basin. The graph indicates that the odor was approximately 90-95% less in the treated slurry compared to the raw manure. The downwind observations during the field application reflected the same results as in  
5 the storage basin.

#### Conclusion

From the foregoing, it is seen that the process of this invention will permit manure comprised of liquid and solids  
10 to have the odors thereof substantially stabilized through the adjustment of the pH of the manure to a level of approximately 7.5 to 9.5 wherein the release of the principal odor producing gases hydrogen sulfide and ammonia will be  
15 substantially minimized along with other odor producing gases which release at pH levels beyond this range. This invention therefore will achieve at least its stated objectives.

What is claimed is:

1. A method of stabilizing odors in animal manure,  
5 comprising: obtaining a quantity of animal manure comprising water and solids which contain sources of odorous compounds including ammonia and hydrogen sulfide, substantially maintaining the water content of the manure, adjusting the pH of the manure to a level that will minimize the release of  
10 odorous compounds including ammonia and hydrogen sulfide to the surrounding air from said manure by mixing with said manure an admixture material to obtain a homogeneous mixture, and maintaining the adjusted pH level at a substantially constant value so as to stabilize the release of odorous  
15 compounds from the manure.
2. The method of claim 1 wherein said pH level is in the range of 7.0 to 10.5.
- 20 3. The method of claim 1 wherein said pH level is in the range of 7.0 - 9.5.
4. The method of claim 1 wherein said pH level is adjusted by adding to said manure a material selected from the group  
25 of kiln dust or fly ash.
5. The method of claim 1 wherein said admixture material is selected from the group consisting of lime, cement kiln dust, combustion byproducts and powdered adsorbents containing  
30 activated carbon, and powdered adsorbents containing dry clay.
6. The method of claim 1 wherein said admixture material is added at a ratio of approximately 0.2 to 1.0 parts to 1 part  
35 manure on the dry weight basis of solids in each.
7. The method of claim 1 wherein said pH level is adjusted



to said level by adding to said manure a quantity of alkaline material.

8. The method of claim 1 wherein said admixture is added to  
5 said manure so that portions of the admixture cling to solids to cause them to settle to a level below the water in said manure.

9. A method of stabilizing odors in animal manure,  
10 comprising: obtaining a quantity of animal manure comprising water and solids which contain sources of odorous compounds including ammonia and hydrogen sulfide, substantially maintaining the water content of the manure, adjusting the pH  
15 of the manure to a level that will minimize the release of odorous compounds including ammonia and hydrogen sulfide to the surrounding air from said manure by mixing with said manure an admixture material in an amount of from about 0.25 to about 0.5 parts to one part manure on the dry weight basis  
20 of solids of each to obtain a homogeneous mixture, and maintaining the adjusted pH level at a substantially constant value so as to stabilize the release of odorous compounds from the manure.

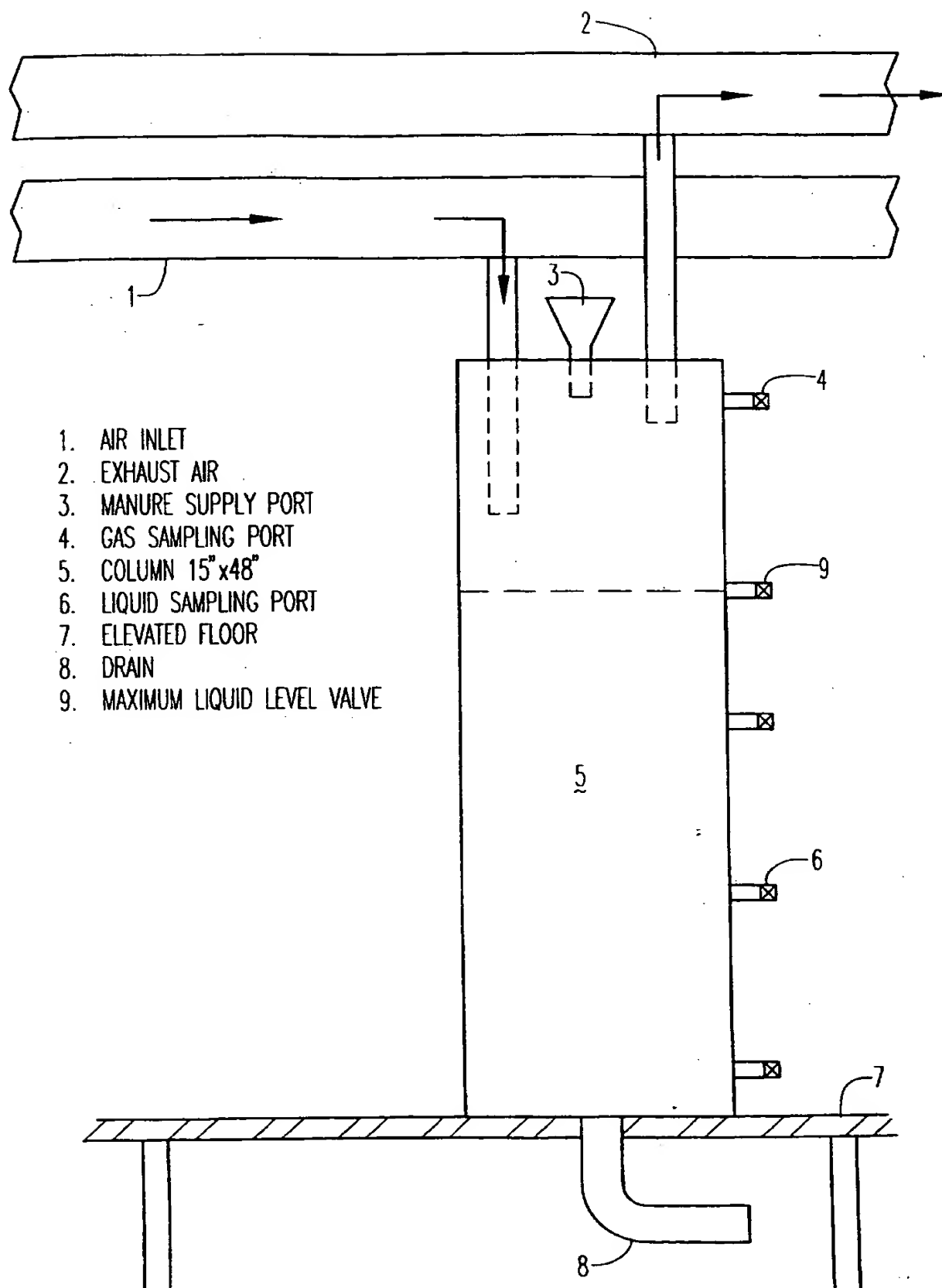


Fig. 8

# INTERNATIONAL SEARCH REPORT

International Application No.  
PCT/US 96/20453

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 C05F3/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 C05F C05D C05B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 201 722 A (SPITZ MICHAEL) 20 November 1986 see claims see column 1, line 54 - column 5, line 18 ---	1-9
X	US 3 877 920 A (CARLBERG GEORGE) 15 April 1975 see claims see column 2, line 4 - line 48 see column 3, line 5 - column 53 see column 4, line 3 - line 14 ---	1-9
X	EP 0 557 078 A (N VIRO ENERGY SYSTEMS LTD) 25 August 1993 see claims 1,3-11,14 see column 8, line 32 - column 9, line 56 --- -/--	1-9

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

11 April 1997

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# INTERNATIONAL SEARCH REPORT

Intern. Application No  
PCT/US 96/20453

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 468 276 A (ROTH TIMOTHY J ET AL) 21 November 1995 see claims see column 2, line 34 - column 3, line 8 ---	1-5
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